

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 12473-6 (1988): Chemical Analysis of Hardmetals by Flame Atomic Absorption Spectrometry, Part 6: Determination of chromium in contents from 0.01 to 2 percent (m/m) [MTD 25: Powder Metallurgical Materials and Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



IS : 12473 (Part 6) - 1988

(Reaffirmed 1999)

Indian Standard

**CHEMICAL ANALYSIS OF HARDMETALS
BY FLAME ATOMIC ABSORPTION
SPECTROMETRY**

**PART 6 DETERMINATION OF CHROMIUM IN CONTENTS
FROM 0.01 TO 2 PERCENT (m/m)**

UDC 669.018.25 : 543.422 [546.76]

© Copyright 1989

**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

Indian Standard

CHEMICAL ANALYSIS OF HARDMETALS BY FLAME ATOMIC ABSORPTION SPECTROMETRY

PART 6 DETERMINATION OF CHROMIUM IN CONTENTS FROM 0.01 TO 2 PERCENT (*m/m*)

0. FOREWORD

0.1 This Indian Standard (Part 6) was adopted by the Bureau of Indian Standards on 25 October 1988, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Chemical analysis of hardmetals by flame atomic absorption spectrometry methods is covered in the following parts :

*Percent Range of
Each Element
(*m/m*)*

Part 1 General requirements	—
Part 2 Determination of calcium, potassium, magnesium and sodium	0.001-0.02
Part 3 Determination of cobalt, iron, manganese and nickel	0.01-0.5
Part 4 Determination of molybdenum, titanium and vanadium	0.01-0.5

*Percent Range of
Each Element
(*m/m*)*

Part 5 Determination of cobalt, iron, manganese, molybdenum, nickel, titanium and vanadium 0.5-2

Part 6 Determination of chromium 0.01-2

NOTE — The method for determination of chromium also permits determination of iron, nickel and manganese within the range 0.01 to 2 percent.

0.3 In the preparation of this standard, assistance has been derived from ISO 7627/6-1983 'Hardmetals—Chemical analysis by flame atomic absorption spectrometry—Part 6 Determination of chromium in contents from 0.01 to 2 percent (*m/m*), issued by the International Organization for Standardization (ISO).

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part 6) specifies the method to be used for the determination of chromium content in hardmetals within the range 0.01 to 2 percent (*m/m*) by flame atomic absorption spectrometry.

1.2 General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in Part 1 of this standard.

2. REAGENTS

2.1 During analysis, only reagents of analytical grade and distilled water or water of equivalent purity shall be used.

2.1.1 Potassium Pyrosulphate

2.1.2 Perchloric Acid ($\rho = 1.54$ or 1.67 g/ml).

2.1.3 Ammonium Citrate Solution — Dissolve 100 g of citric acid in 1 500 ml of water and add 400 ml of ammonia solution ($\rho = 0.91$ g/ml).

2.1.4 Hydrogen Peroxide — 30 percent (*m/m*).

2.1.5 Standard Chromium Solution (1 ml = 1 mg of Cr) — Weigh 2.828 9 g of potassium dichromate previously dried at 110°C and cooled in a desiccator and dissolve in water and transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

3. PROCEDURE

3.1 Test Portion — Weigh, to the nearest 0.001 g, the relevant amount of the test sample indicated in Table 1. Transfer it to a 100 ml conical flask (preferably of quartz).

NOTE — In this special case, the sample shall pass through a 180 μ m sieve.

3.2 Dissolution of the Test Portion — Add 5 g of potassium pyrosulphate and a few drops of perchloric acid to the beaker containing the test portion and heat gently until the test portion is completely dissolved. Add 40 ml of ammonium citrate solution and about 0.5 ml of hydrogen peroxide. Then transfer the solution to a 100 ml polyethylene volumetric flask and dilute to the mark and mix.

3.3 Dilution Volume — Prepare the relevant dilution volume for the analysis according to Table 1 as follows.

3.3.1 Dilution Volume (100 ml = 1 g of Test Sample) — Use the solution in 3.2.

3.3.2 Dilution Volume (500 ml = 1 g of Test Sample) — Transfer 20 ml of the solution in 3.2 to a 100 ml polyethylene volumetric flask and dilute to the mark.

3.4 Preparation of Calibration and Blank Solutions

3.4.1 Prepare at least six solutions according to 3.2 with a matrix composition as similar as possible to the test portion to be analysed but without making up to volume. Then add increasing volumes of properly diluted standard solution of chromium (2.1.5) according to the concentration range to be covered. Make up to 100 ml and mix.

3.4.2 Also prepare calibration solutions with a diluted matrix in accordance with 3.3.2 for dilution volumes of 500 ml, if necessary.

3.4.3 Prepare a blank solution (see 3.4.1) without the addition of chromium.

3.5 Adjustment of the Atomic Absorption Spectrometer — Ignite the flame. Optimize the response of the instrument at the wavelength given in Table 1. Preheat the burner for about 5 min and then adjust the fuel and correct the burner to obtain maximum absorption while aspirating a calibration solution. Make sure that the absorbance reading is not drifting. Aspirate water and set the initial reading to zero absorbance.

3.6 Atomic Absorbance Measurements

3.6.1 Aspirate first the blank solution and then the calibration and test solutions consecutively and record the readings. Aspirate water between each solution. Make at least two measurements for each solution. Solids which build up on the burner slit, must be removed, otherwise they will cause a decrease of sensitivity.

3.6.2 Prepare a calibration curve by plotting the obtained absorbance values of the calibration solutions corrected for the blank against the concentration, in ml per litre, of chromium.

3.6.3 Convert the absorbance values of the test solutions corrected for the blank to mg of chromium per litre by means of the calibration curve.

4. TEST RESULTS

4.1 Calculation — The chromium content, expressed as a percentage by mass, is given by the formula:

$$\frac{C \times V}{10^4 \times m}$$

where

C = concentration, in ml per litre, of chromium in the test solution;

V = dilution volume, in ml; and

m = mass, in g, of the test portion.

TABLE 1 TEST PORTION, INSTRUMENTAL PARAMETERS AND CHARACTERISTICS OF CALIBRATION FUNCTIONS

(Clauses 3.3 and 3.5)

CONTENT	TEST PORTION	DILUTION VOLUME (V) FOR THE TEST PORTION*	OXIDANT	WAVE LENGTH	RECIPROCAL SENSITIVITY FOR PERCENT ABSORPTION*	LINEAR RANGE*
PERCENT	g	ml		nm	μ g/ml	PERCENT
(1)	(2)	(3)	(4)	(5)	(6)	(7)
0.01 to 0.1	0.500	100	N ₂ O	357.9	0.11	0 to 0.12
0.1 to 0.5	0.500	500	N ₂ O	357.9	0.11	0.1 to 0.5
0.1 to 0.5	0.100	100	N ₂ O	357.9	0.11	0.1 to 0.5
0.5 to 2	0.100	500	N ₂ O	357.9	0.11	0.4 to 2

*Guideline for information only.

4.2 Permissible Tolerances — The deviations between three independent determinations shall not exceed the values shown in Table 2.

TABLE 2 PERMISSIBLE TOLERANCES

CONTENT PERCENT (1)	RANGE PERCENT (2)
From 0.01 to 0.20	0.012
Over 0.20 to 2	$0.06 \times (\text{content})$

4.3 Final Result — Report the arithmetical mean of acceptable determinations rounded to the nearest value as shown in Table 3.

4.3.1 If the chromium content is below 0.01 percent, report it as less than 0.01 percent.

TABLE 3 ROUNDING OFF RESULTS

(Clause 4.3)

CONTENT PERCENT (1)	ROUND TO THE NEAREST PERCENT (2)
From 0.01 to 0.20	0.01
Over 0.20 to 2	0.05

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 3310131, 3311375

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg,
NEW DELHI 110002

Telephone
3310131, 3311375

*Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola,
CALCUTTA 700054

362499

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036

21843, 31641

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113

412442, 412519, 412916

†Western : Manakalya, E9 MIDC, Marol, Andheri (East),
BOMBAY 400093

6329295

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001

26348, 26349

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road,
BANGALORE 560158

384955, 384956

Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar,
BHOPAL 462003

66716

Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002

53627

53/5, Ward No. 29, R. G. Barua Road, 5th By-lane, GUWAHATI 781003

33177

5-8-56C L. N. Gupta Marg (Nampally Station Road),
HYDERABAD 500001

231083

R14 Yudhister Marg, C Scheme, JAIPUR 302005

63471, 69832

117/418 B Sarvodya Nagar, KANPUR 208405

216876, 218292

Patliputra Industrial Estate, PATNA 800013

62305

T.C. No. 14/1421, University P.O., Palayam, TRIVANDRUM 695034

62104, 62117

Inspection Offices (With Sale Point):

'Pushpanjali', First Floor, 205A West High Court Road, Shankar Nagar
Square, NAGPUR 440010

25171

Institution of Engineers (India) Building, 1332 Shivaji Nagar,
Pune 411005

52453

*Sales Office in Calcutta is at 5 Chowringhee Approach, P.O. Princep Street,
Calcutta 700072

276800

†Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007

896528

‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, Bangalore 560002

223971